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T.R.E. MEMORANDUM

No. 275

THE PREPARATION OF CARBON RESIN FILMS FOR PRINTED
WIRING RESISTORS

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UNCLASSIFIEDTHE PREPARATION OF CARBON RESIN FILMS FOR PRINTED
WIRING RESISTORSSUMMARY

Diffioulty has been experienced in obtaining resistance film sheet material from resistor manufacturers for printed circuit application. A survey has been made of the problems involved in producing the required resistor sheet in the laboratory, and the memorandum outlines methods and materials required to carry out further experimental work.

1. INTRODUCTION

The use of carbon or graphite, in powdered or colloidal form with a synthetic resin binder, as a resistance element for volume control tracks is common practice in the radio industry. The use of such track material as the resistor element for printed circuit techniques has been proposed in Technical Note No. 43 - Aspect Ratio Resistors for Deposited and Printed Circuits. When uniformly coated sheets are used in this way, or when the resistors are sprayed in position on a printed panel, a suitable resistor varnish is required. Most commercial organisations making carbon film potentiometers have developed their varnishes over a number of years and can prepare mixtures that are practically repeatable. Their formulae are the result of considerable experience and it is evident that there are many variables, not only in the ingredients, but also due to the atmospheric conditions under which the mixtures are prepared and methods of application of the film. A number of formulae that have been developed are given in the Section 2 of this Memorandum. The third part of the Memorandum discusses some methods of applying the varnishes. The electrical characteristics are not discussed since they are similar to those of the carbon film volume controls.

2. RESISTOR PAINT FORMULAE AND SPECIFICATION

The synthetic resin used as a binding medium for the conductive parts of the mixture can be obtained prepared in the form of an oil bound varnish or as a pure oil free resin varnish. These varnishes can be diluted with solvents so that the conductive particles can be dispersed by ball milling. Alternatively the conductive portion can be obtained as a dispersion in the solvent ready for diluting the synthetic resin.

2.1 Oil based synthetic resin varnish

Since the commercial synthetic resin lacquers are not normally supplied to a rigid specification, it is found that the composition varies considerably from batch to batch. In order to reduce the variables in the process it is advisable to have the binder made up to a specification. A typical formulation for an oil based resin has been recommended and the details are given below:-

		Parts by weight
Resin - 1	Pure Phenolic, oil reactive heat hardening resol.	100
Resin - 2	Modified phenolic resin consisting of Resin/Glycerol Esters modified with a diphenylolpropane formaldehyde resol.	100
Drying Oils	Tung oil (China wood oil)	200
Driers	Cobalt Naphenate	5
	Lead oxide	12
Solvents	Xylene	500

The binding medium is prepared by dissolving the pure resin in the drying oil at about 200°C. The lead oxide is added as a thin oil bound paste at 240°C and dissolved. The modified resin is then added at the same temperature. About 100 parts of high conductivity acetylene black is ground into the binding medium with a ball mill, adding the solvent as required. The mixture requires about 48 hours milling. The liquid driers (cobalt naphenate) is added during this stage. The resistor paint can be applied by one of the methods described in Section 3 and the completed resistor should be stoved at about 150°C. A protective varnish coating of the same composition as the binding medium should be applied to the exposed surface of the resistors.

2.2 Oil free synthetic resin varnish

The comparatively new, oil-free compound resin, Araldite, can be used as a binding medium for a resistance paint. Carbon black, powdered graphite or colloidal graphite are ground into the resin in the usual way. The composition of the varnish depends on the method of applying the film to the insulating surface. A typical formula for spraying is as follows:-

Araldite resin	985 E	approx.	350 parts by weight
Carbon black			10-200 parts

The following Solvents are required:

Diacetone alcohol	420 parts
Toluene or Xylene	120 parts
Methyl or Ethyl Acetate	60 parts

The quantities and solvents for application by rollers vary considerably from the above.

In the case of steel rollers the recommended varnish is approximately:

500 parts (wt)	Araldite 985E
560 parts	Methyl or Ethyl Acetate
85 parts	Toluene
82 parts	Diacetone alcohol.

The suggested formulae for synthetic rubber rollers is approximately:

555 parts by weight	Araldite 985E
385 parts by weight	Diacetone alcohol
60 parts by weight	Methyl Ethyl ketone.

The suggested formulae for application by gelatine rollers is approximately:

700 parts by weight	Araldite Resin 985E
115 parts by weight	Tetrahydronaphthalene (Tetraline)
115 parts by weight	Xylene
70 parts by weight	Diacetone alcohol

In each case the required quantity of conductive media must be added and dispersed in the varnish in a ball milling or a colloidal grinder. The completed resistor film has to be stoved at between 160°C for 40 to 80 minutes.

2.3 Colloidal graphite dispersions

Resistance films have also been prepared satisfactorily from colloidal graphite dispersed in the following solvents and added in suitable proportions to the appropriate synthetic resins.

Dispersion medium

Acetone
Naphtha
Water
Xylene
Benzene
Complex solvent

Synthetic Resin

Ethyl Cellulose
Polystyrene
Cresols
Phenol Formaldehyde
Silicone Resin
Ethoxylene Resin

Stoving temperature and time depends on the synthetic resin that has been used. Similar synthetic resins have been used in both Germany and America for the production of carbon film type resistors and volume controls. Some typical formulae are given below and these have been abstracted from the references given.

2.4 Formulae for Carbon-resin Resistor films used in Germany and U.S.A.

2.4.1 "Sator" volume Control Tracks (see B.I.O.S. Report No. 567)

1.2 kg. Carbon black and 2.6 kg. Bakelite lacquer (German No. 362) dissolved in 980 cc of equal parts of ethyl alcohol and benzene. The whole was ground in a porcelain ball mill with silica balls for 200 hours. The mixture was modified by adding bakelite lacquer and solvent until the required value was obtained. It is necessary to ball mill after each addition. The resistance elements were stoved at 180°C for 3 hours.

2.4.2 "Freh." Volume Control Tracks (B.I.O.S. 567)

50 grams of carbon black to 500 grams Bakelite varnish for 1 megohm element.

The mixture was passed twice through a colloidal grinder, once at 20 minutes per litre, then at 40 minute per litre. Stoving was also carried out in two operations, once at 90°C for two hours then at 130-140°C for 40 minutes.

2.4.3 N.B.S. Resistor Paint Formulae

N.B.S. Circular 468

R	Thickness	Pigment	Binder	Solvent	Stoving Temp.
1000	.003 in.	38% Graphite	62% Silicone		275°C
2000	.003 in.	3% Carbon Black	70% Silicone resin		275°C
5000	.003	27% Graphite	77% Silicone resin		275°C
25000	.003	4% Carbon	17% Phenolic	33% Xylene	175°C
		19% Graphite			
		12% Carbon			
		38% Graphite			
25K to 50K	.0015 to .003	7% Carbon black	72% Silicone resin	21% Benzene	275°C
50K to 1 meg.	.001 to .004	11% Carbon Black	66% Ethyl Cellulose		50°C
		2% Graphite			

Ball milling of mixture is necessary and should be carried out for at least 72 hours.

3. APPLICATION OF RESISTOR PAINTS

There are two principal methods of applying synthetic resin films to insulating materials suitable for resistor deposition. These methods are:

- (a) Spray coating by means of compressed air jet.
- (b) Printing, including roller and capillary coating methods.

3.1 Spray coating resistor paint

The application of resistor paint by means of a spray gun is the method most commonly used commercially and the following are among the factors which have to be taken into account to assist in obtaining a repeatable film.

- (1) The distance of the gun from the insulating material.
- (2) The speed of travel of the base material relative to the gun.
- (3) The air pressure applied to the gun and the opening of the controllable jet.
- (4) The size of the fixed jet.
- (5) The shape of the jet of paint.
- (6) The position of the resistor area relative to the line normal to the axis of the jet.
- (7) The viscosity of the paint.
- (8) The temperature of the paint.
- (9) The temperature of the air and the insulating base.
- (10) The humidity of the atmosphere through which the paint passes.
- (11) The number of coats of paint applied over the area.
- (12) The level of the paint mixture in the spray gun container and how long the paint has been there.

Variations in the composition and the constituents of the paint itself and stoving time and temperature provide at least as many variables, but these would be common to other methods of application. Several of the factors listed can be automatically controlled and the technique has been so well developed by the industry that standard deviations of the order of 5% have been achieved in production (see appendix 1). Spray guns are arranged at fixed distances from the work and the panels to be sprayed, pass by at a constant speed. The spray guns are automatically switched on just before the work passes in front. The position of the resistor area relative to the line normal to the axis of the jet introduces what can be termed an attitude factor and in some cases it is necessary to allow for this when laying out the resistor panel. When an engraved meander is used, this attitude factor is not so important as when the aspect ratio method is used.

3.2 Printing resistor ink

Printing of varnishes from rotating rollers is also a well established commercial practice; gummed paper and transformer interlocking paper are both coated in this manner. Steel, rubber and gelatine rollers are used and formulae for suitable varnishes are listed in Section 2. Another method is the capillary coating used on some commercial vacuum controls. The card material passes under the end of a light capillary syphon and a film of carbon and resin is drawn out over the card. The clearance between the syphon and the card determines the thickness of the deposited film. It is usual to maintain this distance constant and to obtain the required resistance range by the composition of the binder and conducting pigment. To deposit separate resistors a number of capillary stencil pens such as the largest Uno type could be used.

The design of a suitable printing machine to coat cards say six inches or more wide would entail a considerable amount of development work, but a machine to deposit separate resistors using stencil pens could be made comparatively simply.

The factors affecting the ability to repeat results are not so numerous as those involved in the spraying operation and it has been noticed that the films deposited in this manner are more uniform in

appearance. The accuracy of the method depends on the design of the printing machine and the uniformity of the resistance mixture. The humidity, temperature and altitude factors are not so important as in the spraying method. The roller printing method necessitates covering one or both sides of the sheet with resistance ink and then removing this from parts of the sheet by light sand blasting, scratch brushing, or by chemical means, thus forming the aspect ratio resistors.

4. CONCLUSIONS

To undertake the preparation of carbon resin films on insulating panels several courses of action are possible. The oil based resins require considerably more apparatus and effort to prepare than the oil free resins. Commercially available varnishes could be used to see if the oil based varnish has any particular merit, otherwise it is recommended that laboratory work should be confined to the oil free resins.

The apparatus required for spraying volatile liquids is both cumbersome and complex to operate but it is possible to obtain reproducible results. The effort involved is not considered justified in view of the comparative simplicity and clean operation of a printing machine. More development effort would be required for the design of an efficient printing machine than for the erection of a spray plant, but it is considered that greater control over the process would be obtained from a capillary or roller printing machine.

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APPENDIX 1

SELECTION TOLERANCE ON SPRAYED VOLUME CONTROL TRACKS

Some variable resistors were required for use as pre-set controls in an equipment where space was limited. The tracks from commercial miniature potentiometers were obtained for assembly directly on to a terminal panel. Since it was known that the particular manufacturer used an automatic spray plant, the tracks were measured on receipt to ascertain the distribution of the resistance values. The nominal tolerance was given as $\pm 20\%$.

The first batch was of 51 samples of a nominal 25,000 ohms. The average resistance value of the samples received was 24.2K ohms. The standard deviation from this was 0.548K ohms or 2.6%. The range in resistance value was 22.6K to 25.4 or from -6.6% to +4.9% of the average.

The second batch consisted of 50 samples of again a nominal 25K ohms and this time the average value was 25.56 ohms. The standard deviation was 1.68K ohms or 6.6% and the resistance range from 22.2K to 28.2 K or -13.2% to +10.6%.

The third batch was of a hundred samples of 1000 ohms nominal resistance and the average came out to 910 ohms. The standard deviation from this value was 76.05 ohms or 8.34%. The resistance range in this case was from 700 ohms to 1100 ohms or from -23.2% to +20.6% of the average. These tracks were from a miniature potentiometer and the area was smaller than the earlier batches. The results from the three batches are illustrated in Figure 1 attached.

APPENDIX 2

SELECTION TOLERANCE ON SOME CAPILLARY COATED

RESISTANCE FILMS

Several sheets of volume control track material that had been coated by a capillary method were obtained from Electronics printed on them by the method described in T.R.E. Memo. No. 125.

Six samples each one inch square were made, the average resistance was 574 ohms and standard deviation was 5.8%. In nine samples each half inch square the average resistance was 6.75K ohms and the standard deviation was 3.5%. Eleven samples each quarter inch square were measured, the average resistance was 4.7K ohms and deviation was 15.7%.

Eighteen resistors of various aspect ratios were measured, the standard deviation from the mean ohms per square was 13.5%. Ten resistors of one inch square nominal value 100K per square, average value 90K per square, deviation from Nominal was 13.4%. Deviation from the mean was 9.3%. Ten resistors of nominal 100K square were cut into quarter inch squares. Average resistance 99K, deviation from both nominal and average resistance was 6.6%. Considerably more samples would be required to carry out a correct statistical examination, but these results indicate that a reasonable tolerance on the value required was obtainable by the capillary method.

APPENDIX 3

APPARATUS REQUIRED FOR PREPARING CARBON PIGMENTED RESIN VARNISHES

The following lists show the minimum equipment required to commence experimental work on making carbon resin resistors.

Equipment for oil free resin varnish

Ball mill or colloidal grinder for mixing resins and pigment (about 1 litre capacity).

Drying oven for stoving prepared sheet (thermostat control up to 350°C).

Filters for removing insoluble matter

Electric stirring apparatus

Solvent for keeping apparatus clean

Resins and Solvents.

Additional equipment for preparing oil based synthetic resin varnish

Sand bath heater to raise temperature to 300°C.

Metal kettles for cooking resins in oil

Grinding machine for powdering resins

Resins, oils, driers, and solvents.

Conducting materials

Acetylene or carbon black (Shawinigan Chemicals Ltd.)

Powdered graphite - Messrs. Multiple Acting Flux Ltd.

Colloidal graphite dispersed in solvents (Achesons Colloids Ltd.)

Laboratory apparatus

Weighing machine and weights, measuring flasks

Spatulas, stirring rods and assorted beakers

Filters, filter papers and stands

Thermometers up to 400°C

Clean and drying cloths

Screw top jars (Kilner) for mixtures

Dessicators and crucibles for drying powders

The apparatus required for the application of the resistance film is listed below.

Spray gun application (special room and exhaust fans required)

Dry compressed air supply and pressure control

Spray gun with feed pipes

Assortment of fixed jets for gun

Liquid containers for spray guns

Motor driven turntable to hold masked work

Electrical controlled air valves for controlling compressed air.

Printing method (can be operated in normal laboratory)

Motor driven or hand operated printing machine to be developed, using capillary tubing or rollers.

1st June, 1950

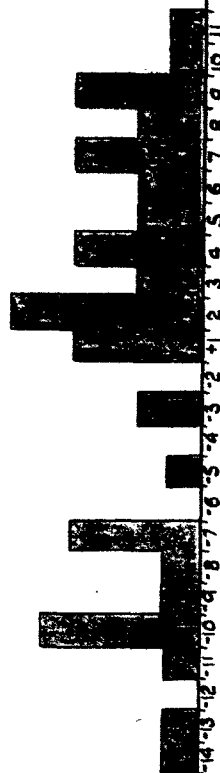
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EACH SQUARE REPRESENTS ONE SAMPLE.

BATCH 1. 51 SAMPLES
NOMINAL 25K TRACKS.



BATCH 2. 50 SAMPLES
NOMINAL 25K TRACKS.



BATCH 3. 100 SAMPLES
NOMINAL 1K TRACKS.

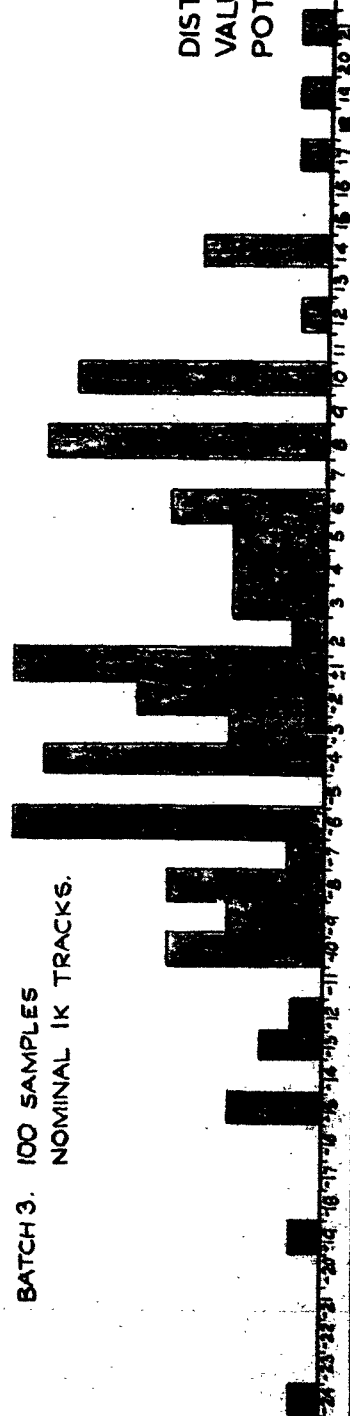


FIG. 1.

DISTRIBUTION OF RESISTANCE
VALUES OF CARBON FILM
POTENTIOMETER TRACKS.



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